

What is claimed is:

1. A method for preventing polymerization of an acrylic acid during a separation of the acrylic acid from an acrylic acid aqueous solution comprising:

5 the acrylic acid aqueous solution contains glyoxal and/or its hydrate;

10 the separation is conducted in an azeotropic dehydration column in the presence of an azeotropic solvent;

15 the acrylic acid, the glyoxal and/or its hydrate are separated from the acrylic acid aqueous solution and withdrawn from the bottom of the column, wherein;

50% or more of the glyoxal and/or its hydrate with respect to 100% of total glyoxal and/or its hydrate contained in the acrylic acid aqueous solution are withdrawn from the bottom of the column.

2. The method according to claim 1, wherein the concentration of water in liquid phases at the 3rd to 6th plate of theoretical 20 plates in said azeotropic dehydration column is 0.1 mass % or more.

25 3. The method according to claim 1, wherein the concentration of acrylic acid in an aqueous phase of the condensate from the top of said azeotropic dehydration column is 0.5 to 5.0 mass % and a bottom effluent withdrawn from the bottom thereof contains 30% or more of acetic acid contained in the acrylic acid aqueous solution fed into said azeotropic dehydration column.

30 4. The method according to claim 1, wherein an azeotropic solvent having a solubility in water of 0.5 mass % or less at room temperature is used.

35 5. The method according to claim 4, wherein said azeotropic solvent is an aliphatic hydrocarbon having a carbon number of

7 or 8 or an aromatic hydrocarbon having a carbon number of 7 or 8.

6. The method according to claim 1, wherein the top temperature
5 of said azeotropic dehydration column is 40 to 50°C and the bottom temperature thereof is 90 to 110°C.